Batch Modeling and Process Monitoring

Geir Rune Flåten

Agenda

- CAMO
- Batch analysis background
- Challenges
- CAMO's approach
- Example
- Alternative strategies
- Demo
- Next Steps

We Develop Multivariate Data Analysis Software & Solutions

- Founded in 1984, we're pioneers and leaders in the field
- Used in 3,000 organizations and by over 25,000 people around the world

DATA ANALYSIS	PROCESS APPLICATIONS	ANALYTICAL ENGINES	SUPPORT & SERVICES
The Unscrambler® X The Unscrambler® X Batch Modeling	Unscrambler X Process Pulse II	The Unscrambler® X Engine	CAMO

CAMO
Bring data to life

We Develop Multivariate Data Analysis Software & Solutions

- Founded in 1984, we're pioneers and leaders in the field
- Used in 3,000 organizations and by over 25,000 people around the world

Design-Expert 9

DATA ANALYSIS	PROCESS APPLICATIONS	ANALYTICAL ENGINES	SUPPORT & SERVICES
The Unscrambler® X The Unscrambler® X Batch Modeling	Unscrambler X Process Pulse II	The Unscrambler® X Engine	CAMO

CAMO
Bring data to life

The CAMO World

PHARMA, PAT/QBD

INDUSTRIAL, CHEMICAL/ENERGY

AGRICULTURE/FOOD/FEED (AFF)

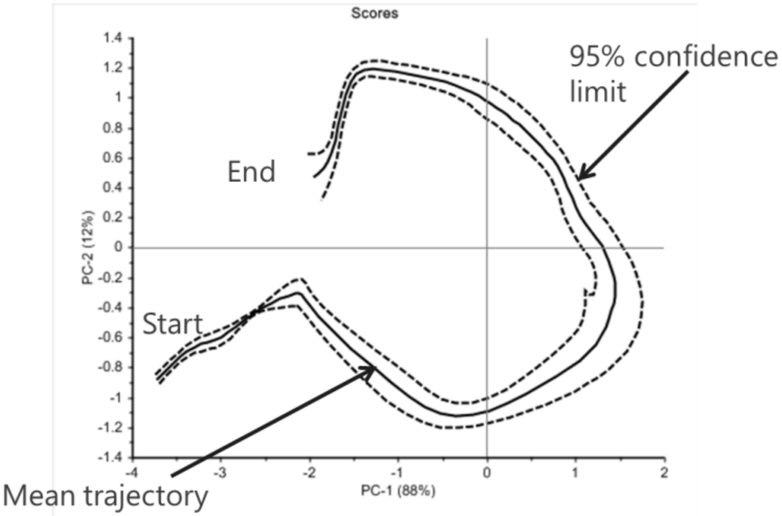
Bring data to life

The CAMO Strategy

CAMO
Bring data to life

Batch - Objective

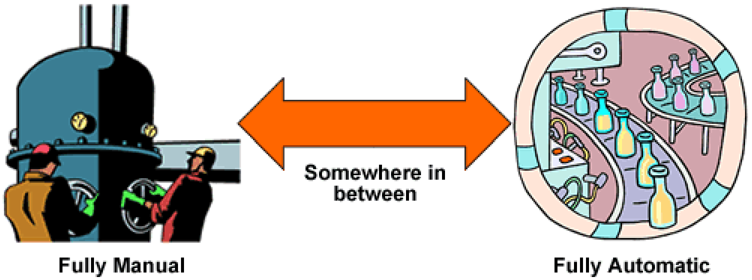
- Real time monitoring
- Real time troubleshooting



Background

Batch definition: Transition from raw materials to product [intermediate]

Batch process control is recipe driven and the operations are in most cases not automatically adjusted to accomodate raw material variations, changes to uncontrolable factors and other circumstances.



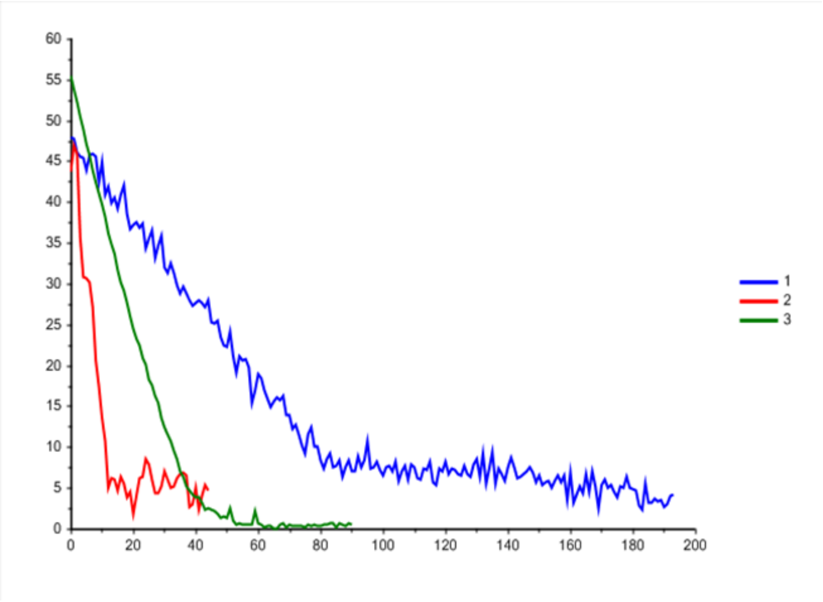
Background – Batch Process Questions

- How can I analyse the batch data from design experiments for process optimisation?
- Are the batches similar?
- Can I find the reason why product quality for some batches lies outside the specifications?
- Are there any effects from raw materials/season/operator/equipment?

- Multivariate Batch Monitoring is important for several reasons:
  - Quality control and event detection
  - Continuous process improvement

Challenge 1: Inequal length and start time

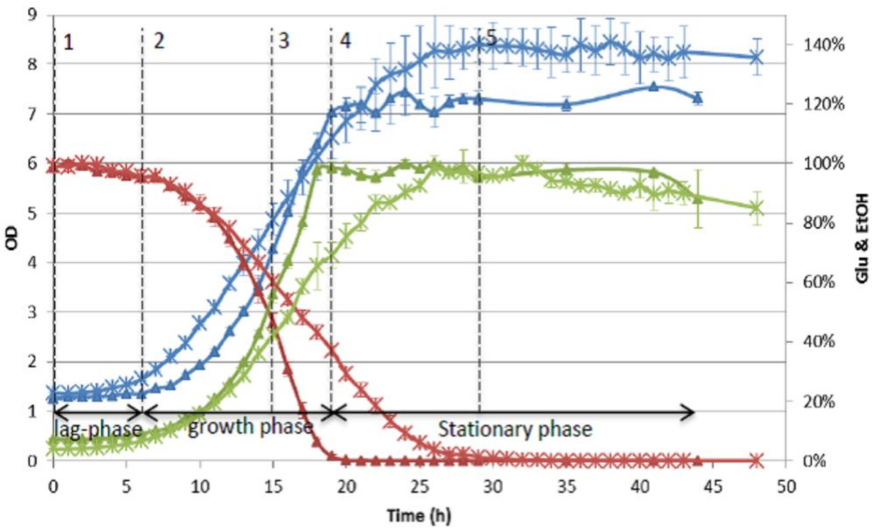
Most batch modelling approaches assume equal lengths of batches:
Same t_0 and the same number of time points for each batch

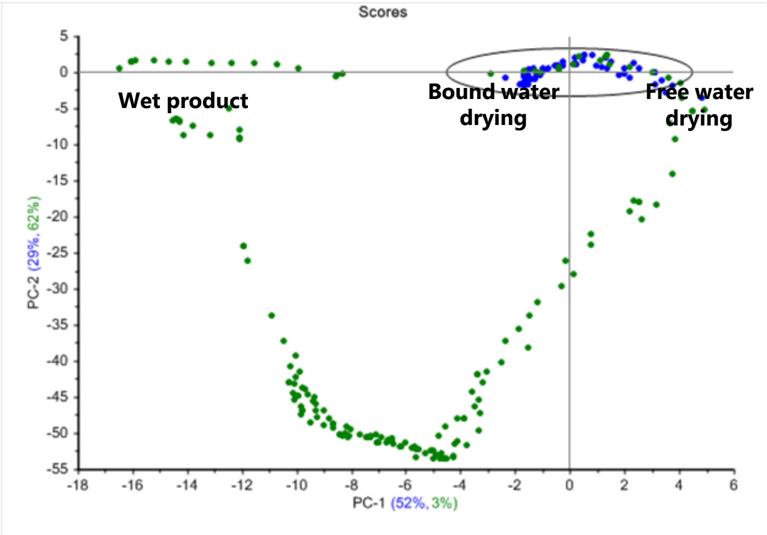


Bring data to life

Challenge 2: Phase transitions and rate changes

Multiphase stages exhibit non-linear system dynamics which makes modelling of phase transitions challenging

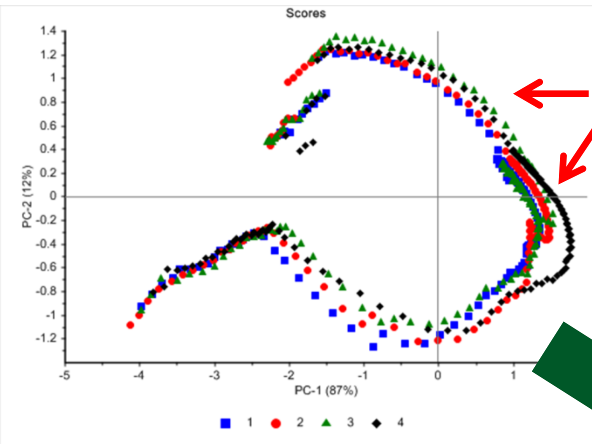




CAMO
Bring data to life

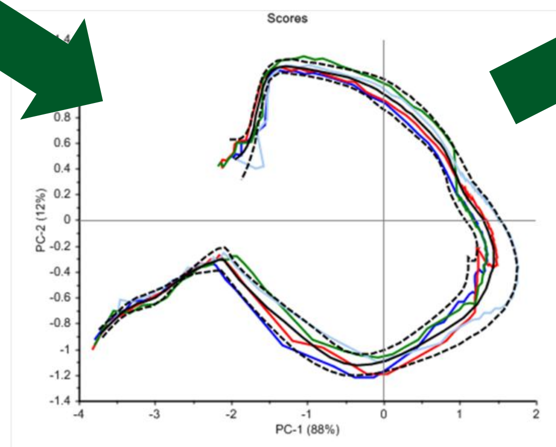
CAMO’s approach

Perform Principal Component Analysis and validate the model across batch

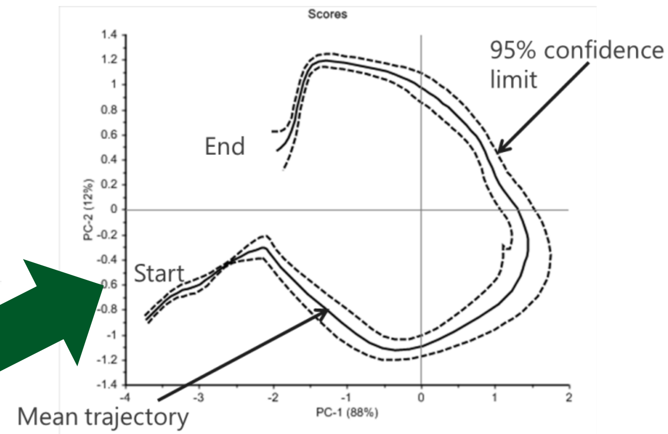


Note the non-linear behaviour!

Score plot of golden batches



Using CAMO’s methodology relative time trajectories are calculated with a new PCA model



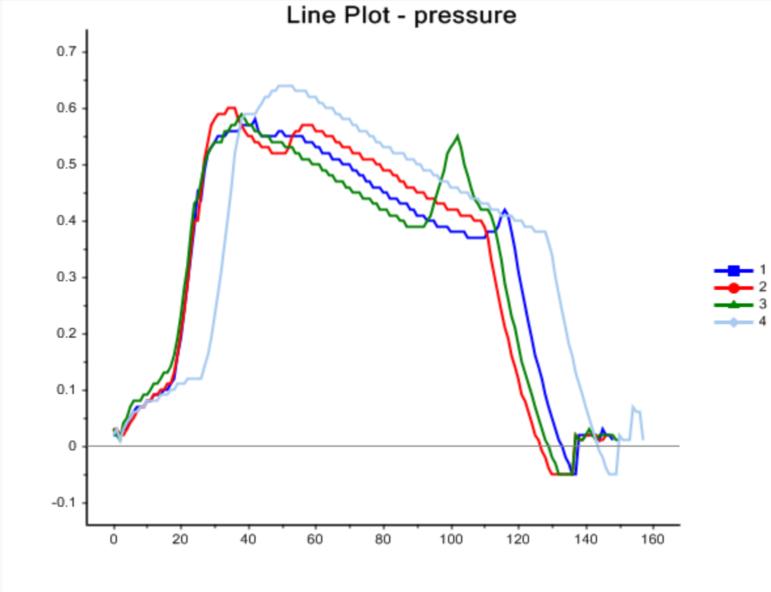
Mean trajectory and dynamic SD limits calculated

CAMO

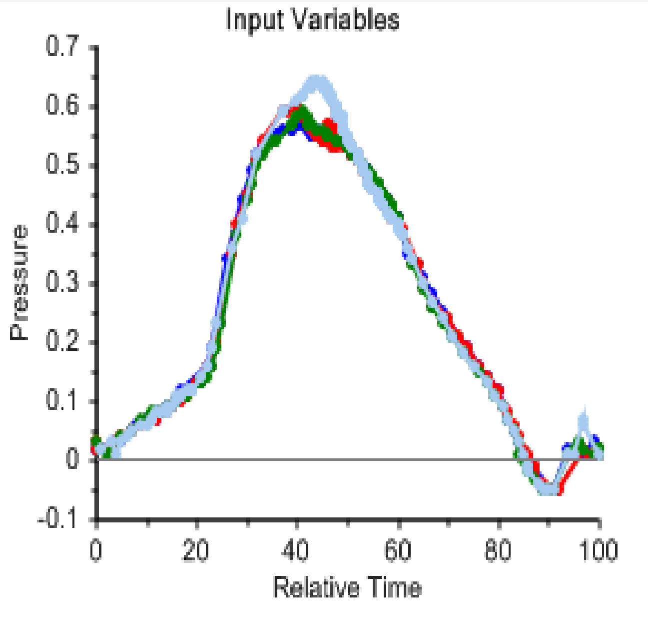
Bring data to life

Visualising individual Process Variables

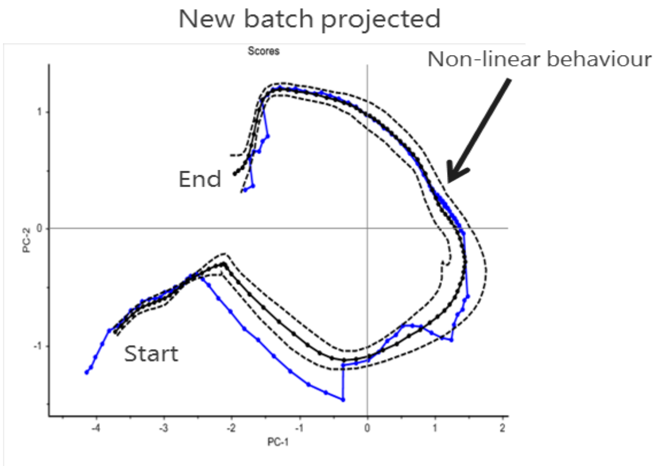
Raw data - Looks like the batches are different

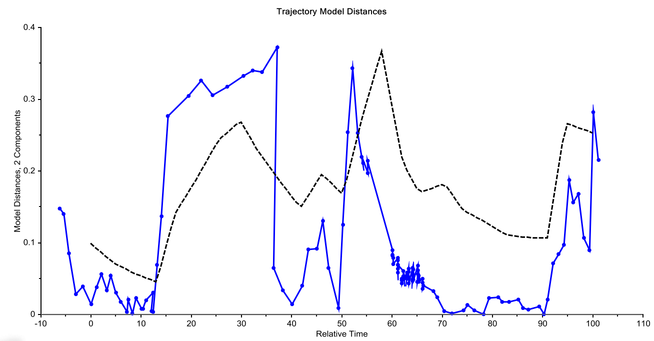


... but in reality: The same trajectory

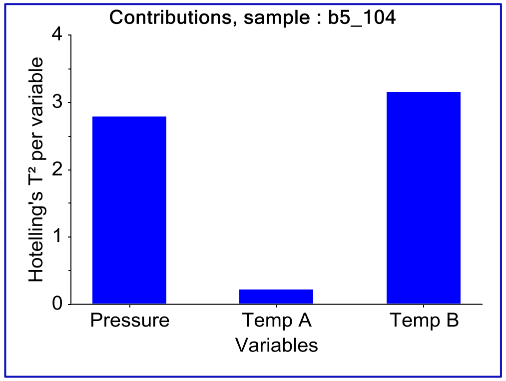


Monitoring a New Batch





- New Batch (**Batch 5**) ran outside dynamic control limits for portions of the process.
- Drill down for sample 104 showed that Pressure and Temp B variables had high contributions in comparison to golden operations for *that relative time*



CAMO
Bring data to life

Method comparison

Scenario	CAMO	Time-wise	Batch-wise
All batches are linear with common start and end	+	+	+
The model shows scores for individual samples	+	+	-
The model requires equal batch lengths	No	Yes	Yes
Historical batches have various relative times	+	Warping?*	Warping?*
Projection of new batch showing non-linear behaviour	+	-	-
New batch has different sampling rate	+	-	-

* Warping may distort the relative time

+ = handled, **-** = not handled

CAMO
Bring data to life

Example Case

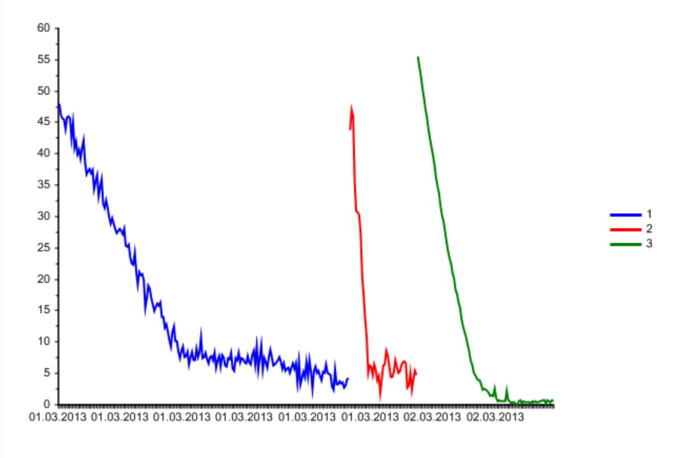
- Chemical reaction
- 3 historical batches
- Three variables: Reactant, intermediate and product (predicted online with a model based on Spectroscopic data)
- PCA on the three batches
- Projecting one new batch

CAMO
Bring data to life

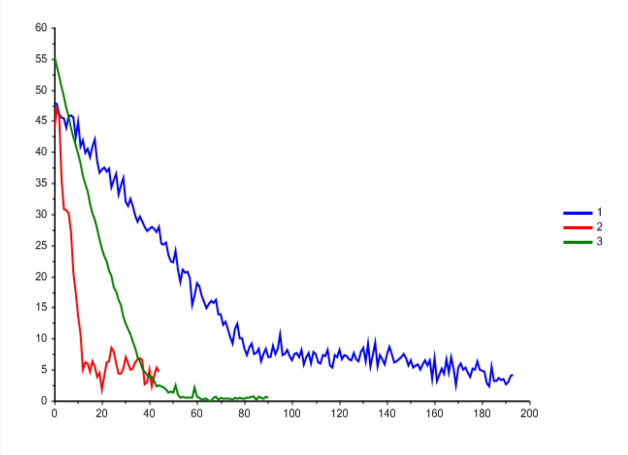
Line plot

Reactant, 3 batches

Consecutive

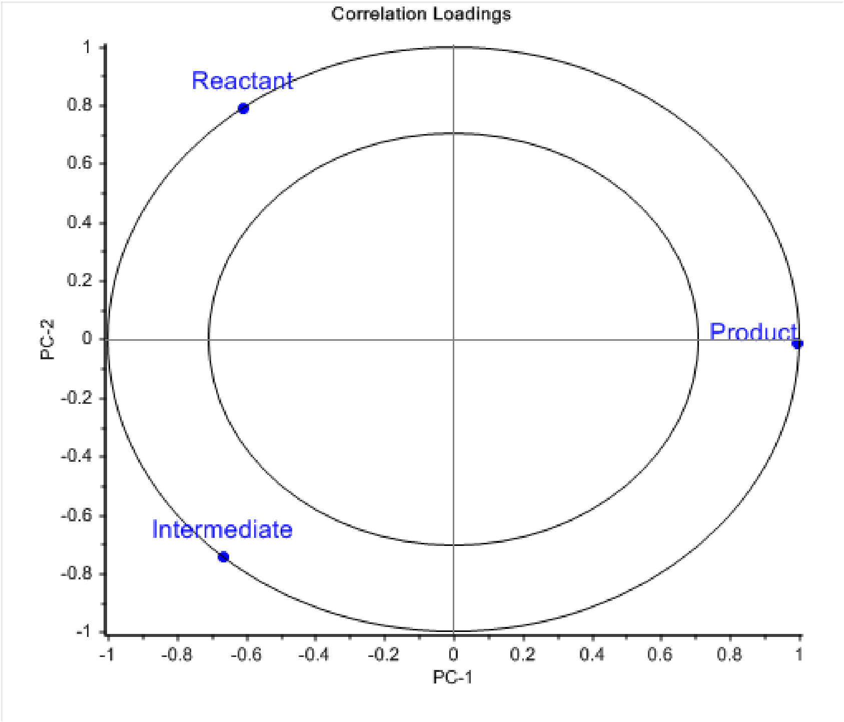


Folded



Correlation loading plot

Not so exiting, but shows how the reaction progresses



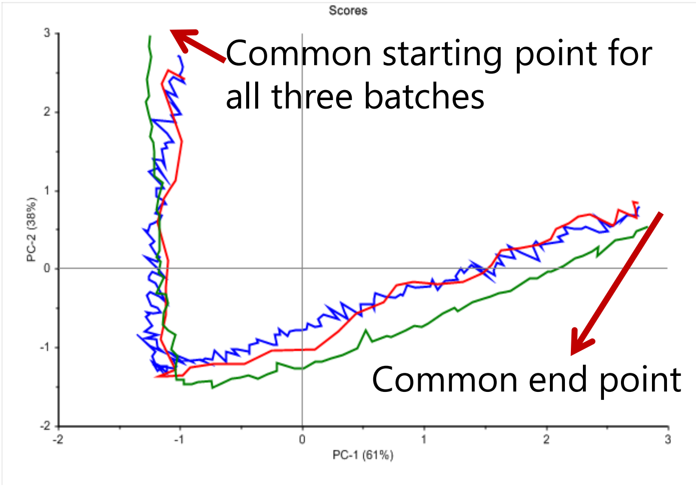
CAMO

Bring data to life

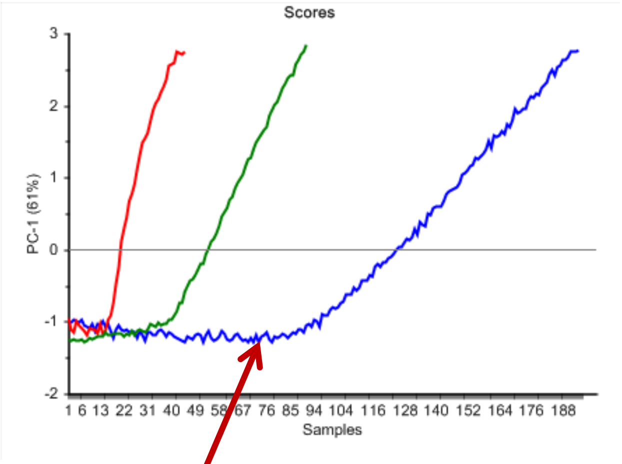
2D score plot– historical batches

Uneven number of data points per batch does not affect the chemical time in the 2D score space

2D score plot

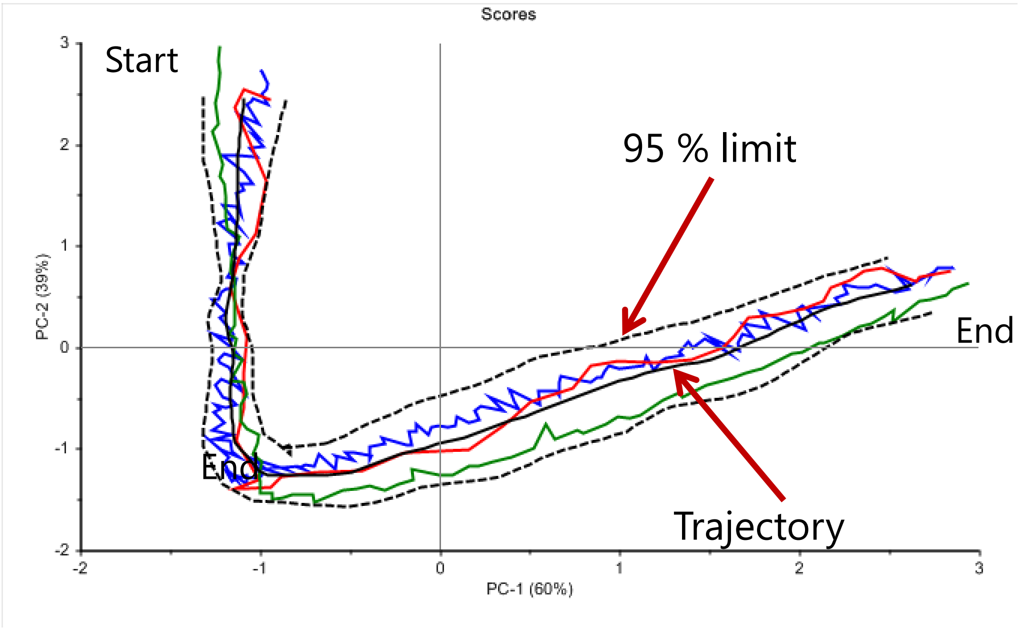


Scores, PC1



Does not reflect the relative reaction time!

2D score plot– trajectory model

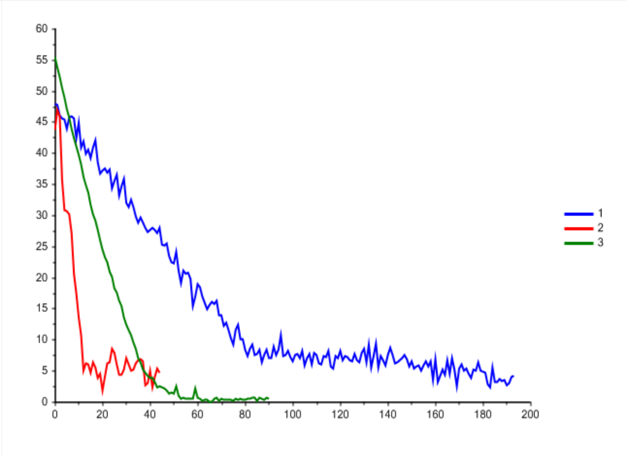


Bring data to life

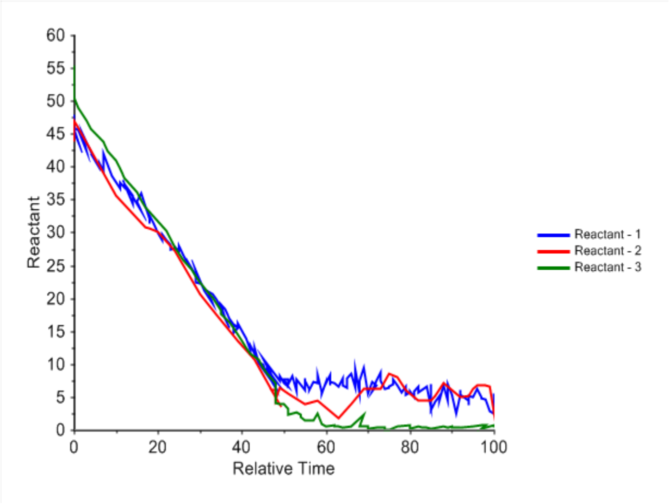
As the method estimates relative time it also enables *individual* variables to be displayed in relative time

Line plot: Reactant, 3 batches

Folded



Relative time

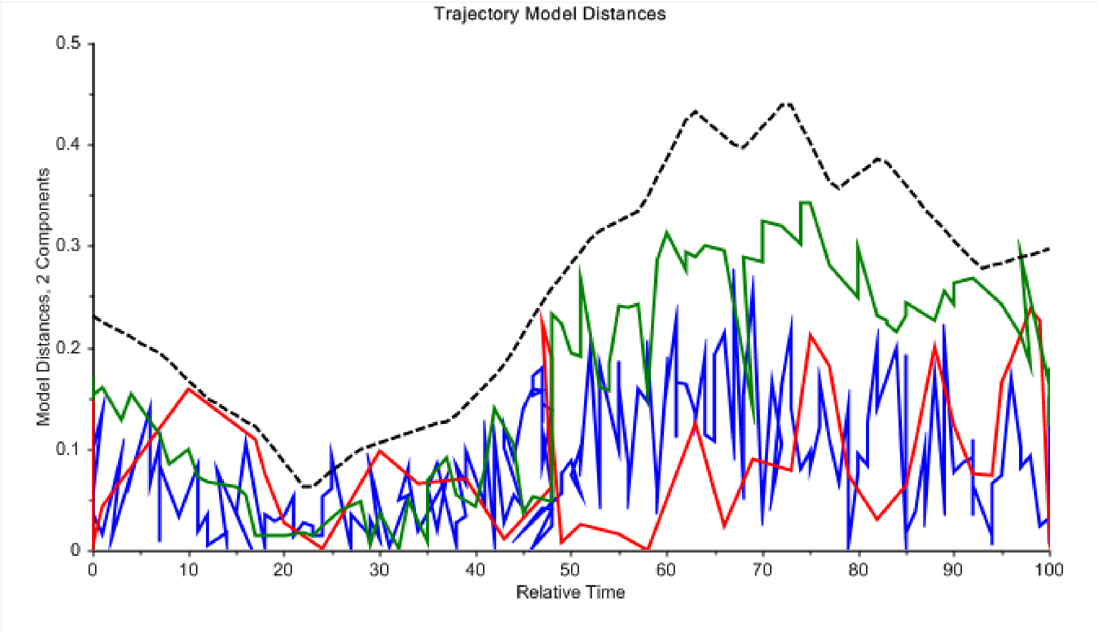


Bring data to life

Trajectory model distance

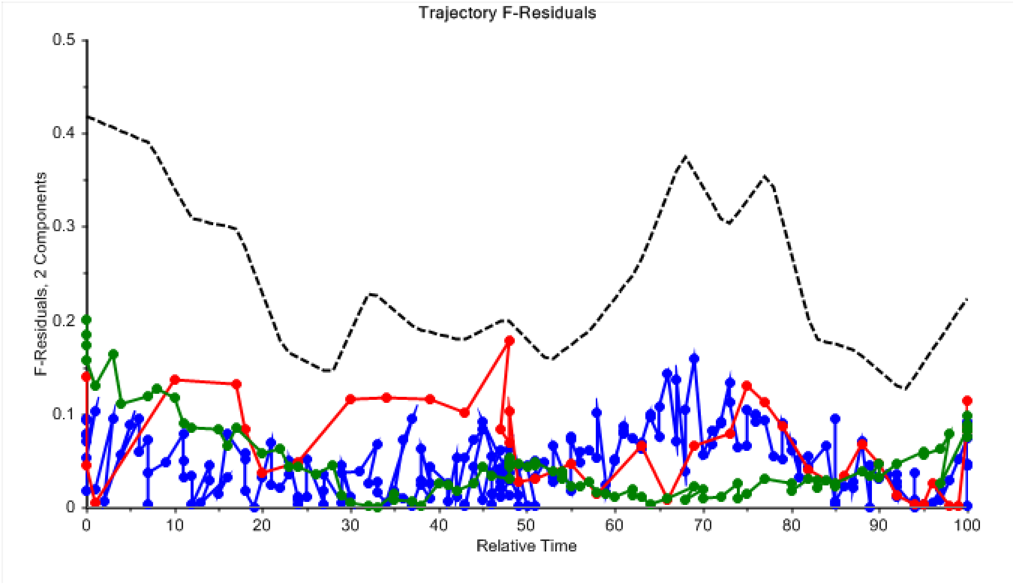
A one-dimensional representation of the limits in the

2D score plot



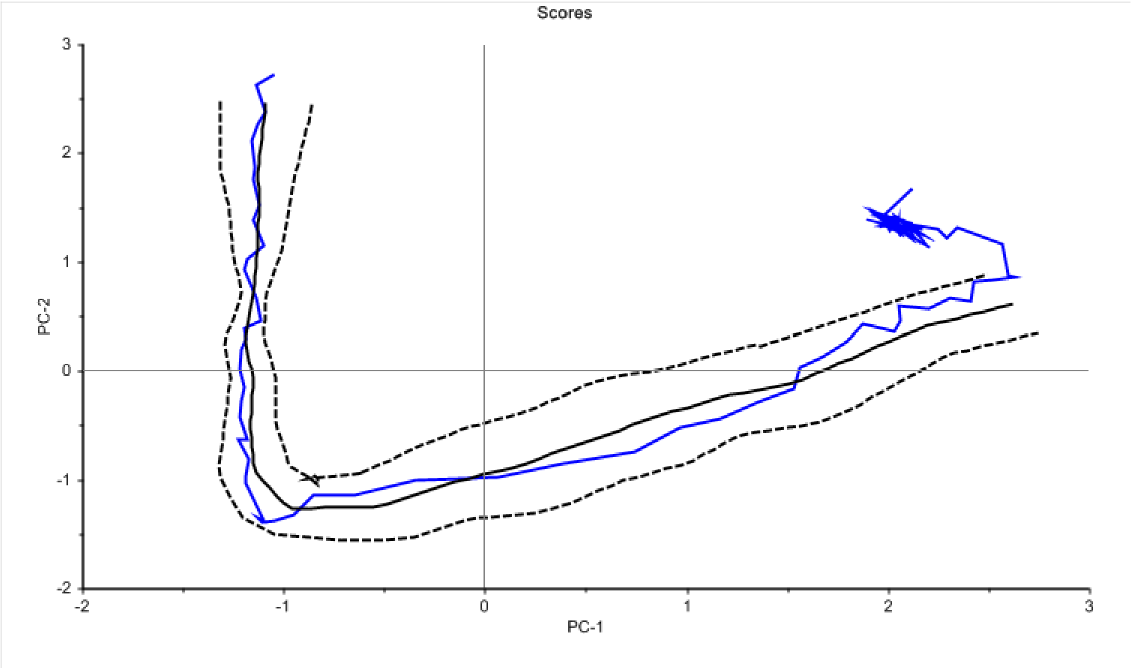
Bring data to life

Trajectory F-Residuals



Projecting a new batch Score plot with limits (95%)

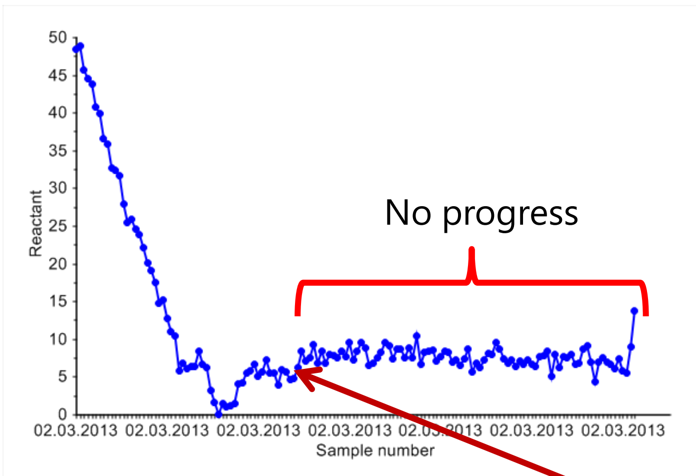
Independent of the sampling rate and number of points

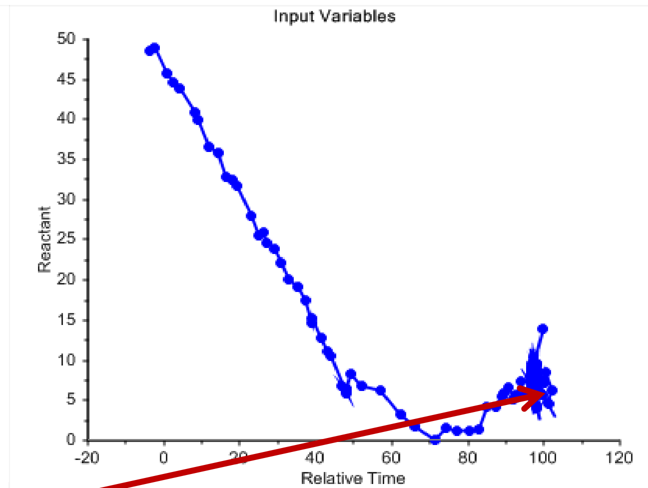


Line plot of the raw data

As sample number

In relative time

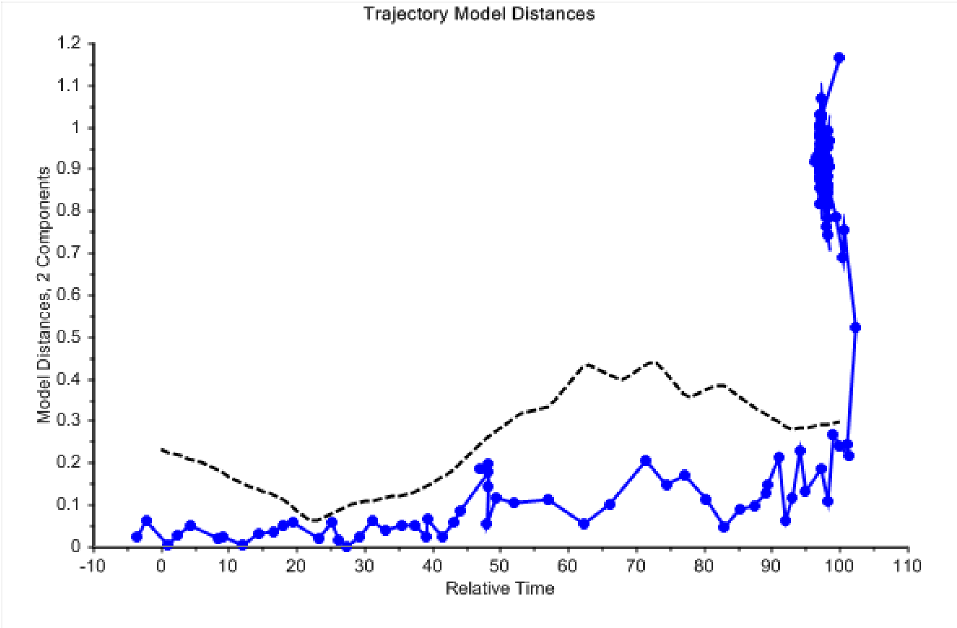




Sample number 55,
reaction is finished

Trajectory model distance

Note how the end of the reaction is visualized correctly due to the relative time axis



Bring data to life



One method for all?

Various approaches depending on application

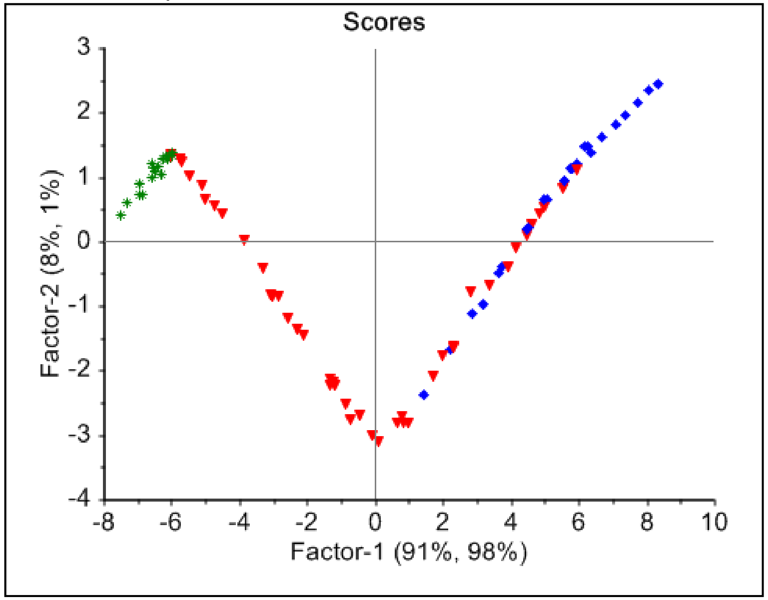
1. Prediction of the yield/quality directly with suitable in-line sensors, e.g. spectroscopy
2. Projecting the new batch onto an endpoint model and decide if the process has reached its end
3. Project the new batch on one existing batch for a qualitative visual assessment
4. Follow the batch progression with a moving-block method; suitable e.g. for mixing processes
5. Project onto a batch model where dynamical limits for distance to model and residual distance have been established from so-called golden batches

Case 1: Direct prediction

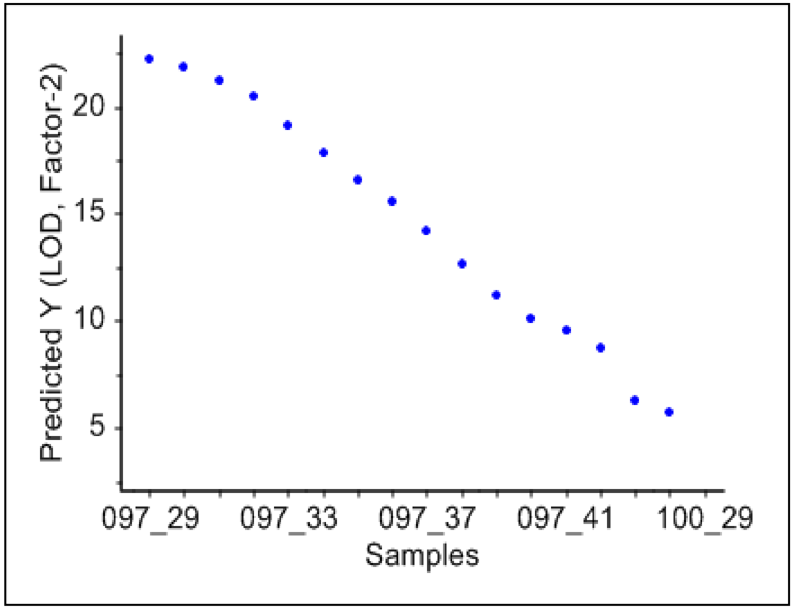
1. Establish a model for prediction of product quality
2. Apply model in real-time

Example: Prediction of moisture in a fluid bed dryer operation with NIR spectroscopy, RMSE; validation over batch = 0.30

Scores with phases of drying in color (Blue = 1, Red = 2, Green = 3)



Predicted values (loss on drying)



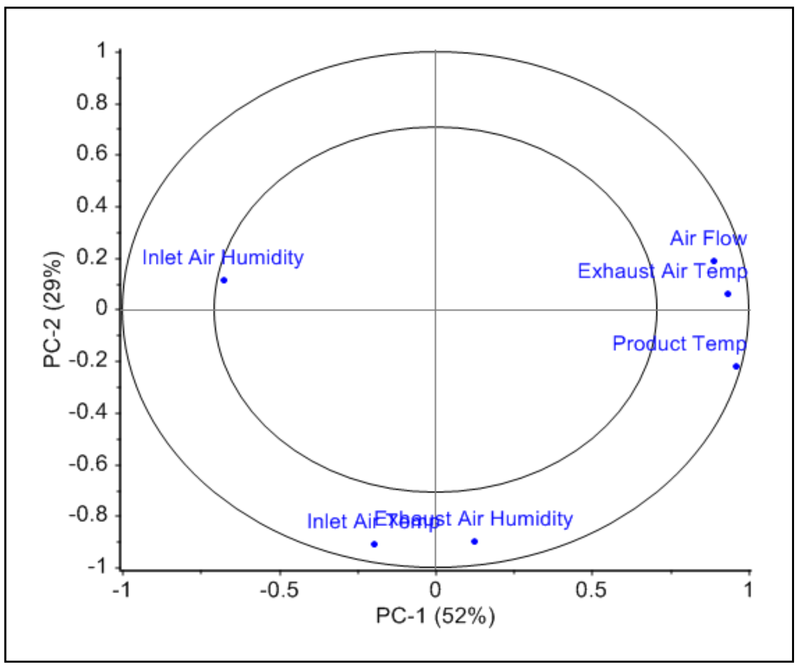
CAMO
Bring data to life

Case 2: Endpoint model

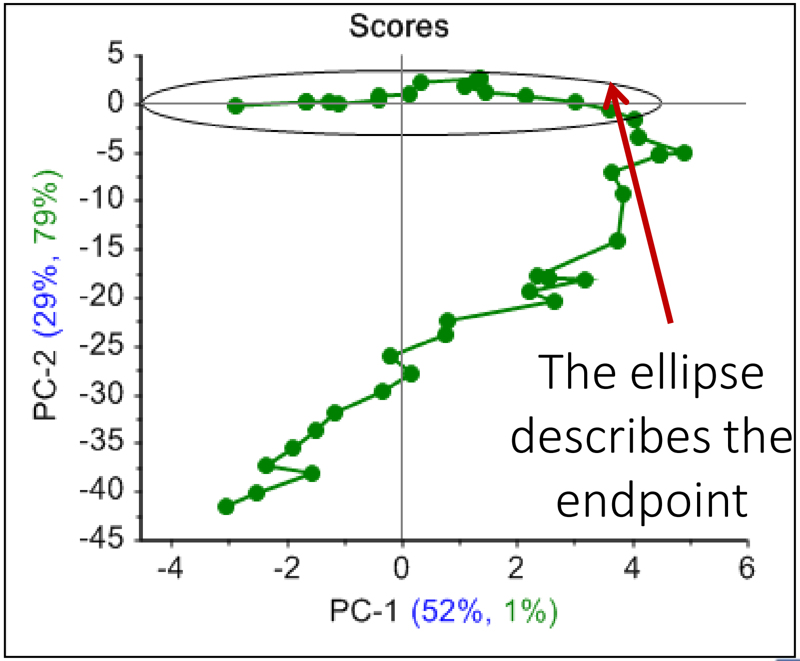
1. Establish a model for the endpoint for a number of good batches
2. Project new observations onto this model

Example: Fluid bed dryer using six process variables

Correlation Loadings



Projected Scores



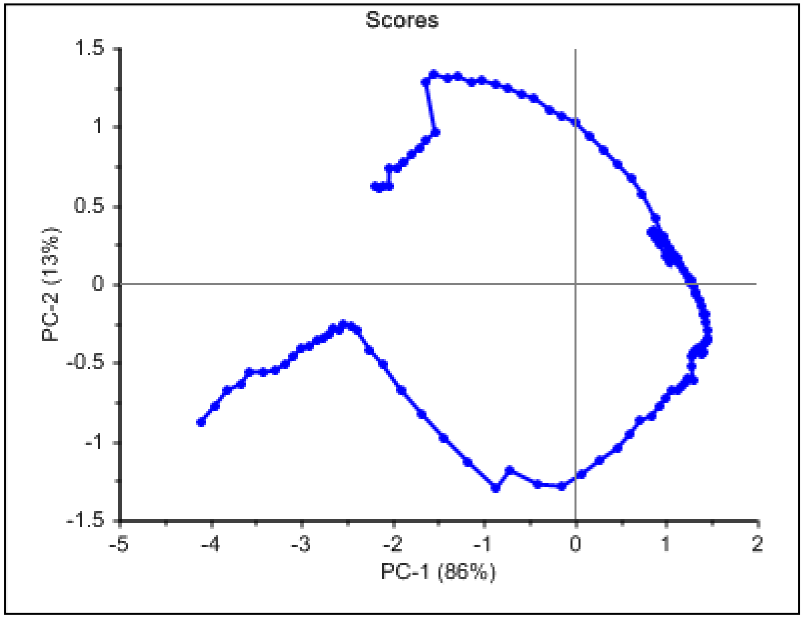
Bring data to life

Case 3: Visual projection

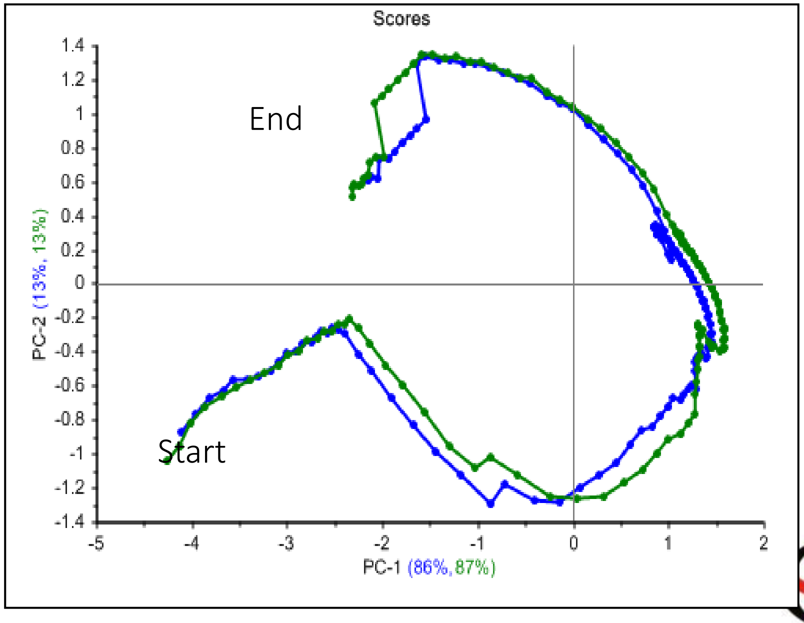
1. Establish a model for the one (or more) batch(es)
2. Project new observations onto this model

Example: Chemical reaction with three variables; Temperature A and B, pressure

PCA for batch 1



Project batch 2



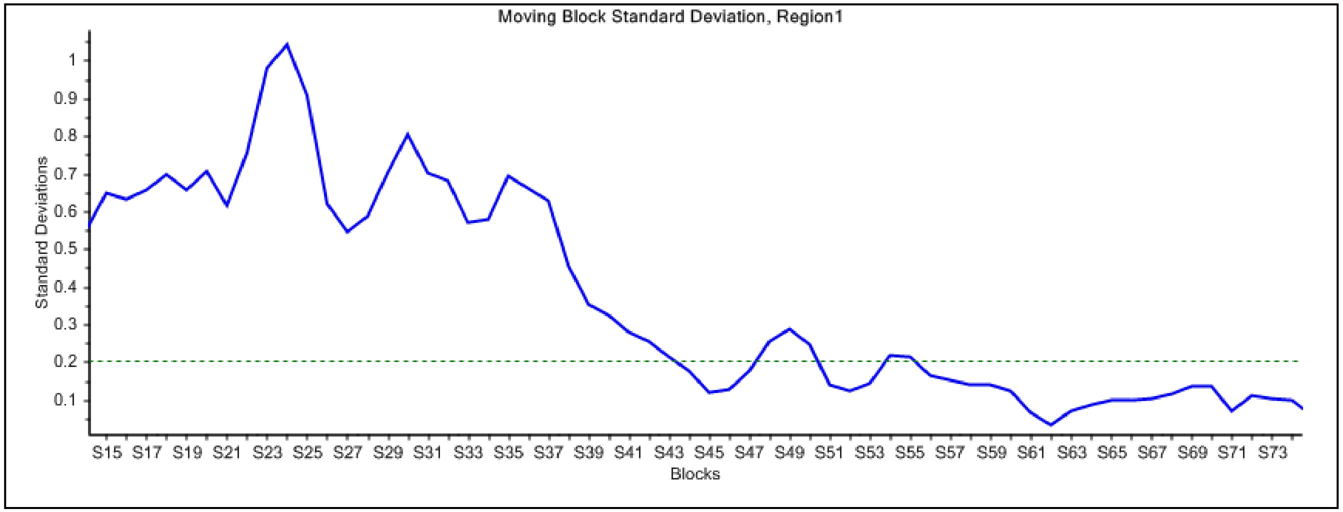
CAMO
Bring data to life

Case 4: Moving block method

1. Establish a moving block model for one batch and set limits for standard deviation, mean value and with an f-test; whatever is applicable
2. Project new observations onto this model

Example: Mixing process with NIR spectroscopy

- Fluid bed dryer operation
- NIR-spectra, 1093 variables



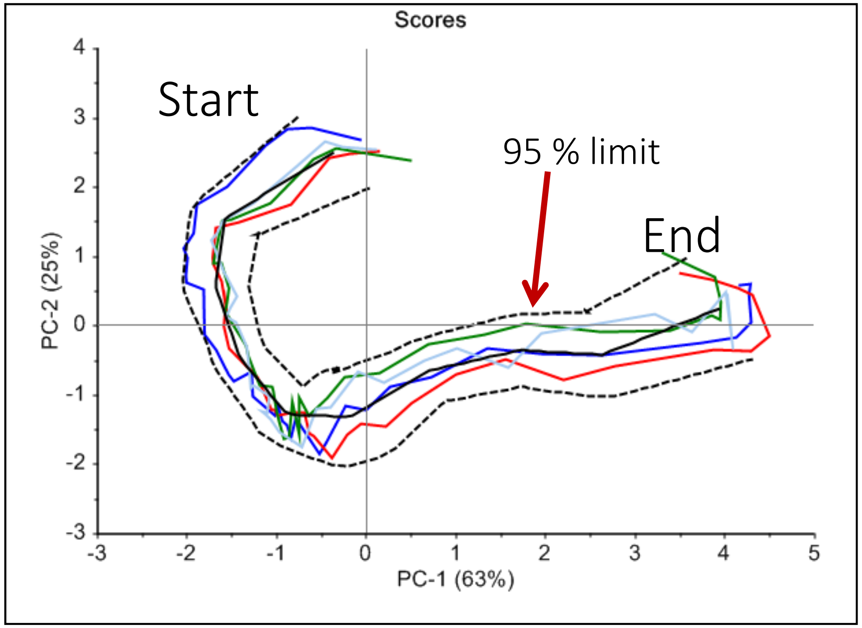
CAMO
Bring data to life

Case 5: Batch model with critical limits

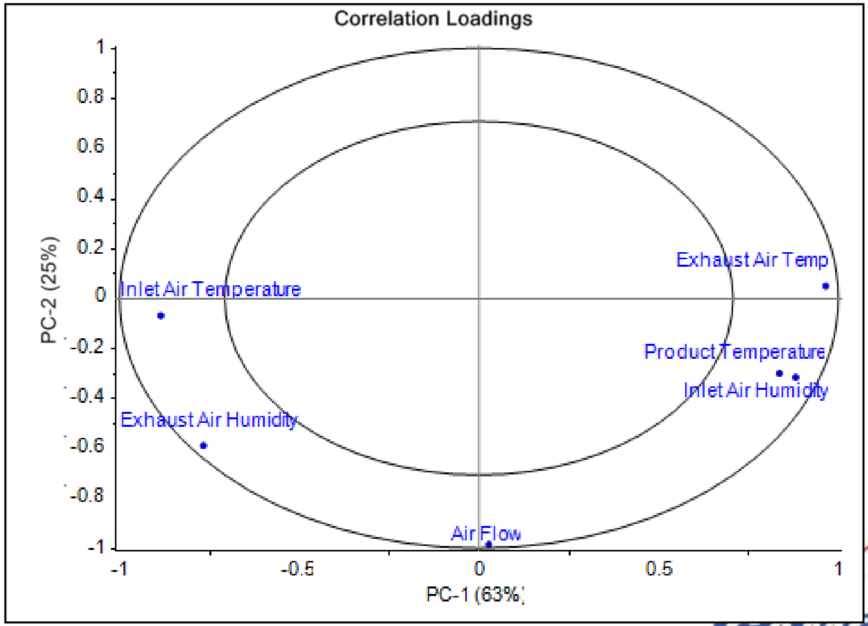
1. Establish a model for golden batches
2. Project new observations onto this model

Example: Fluid bed dryer, six process variables (as above but for the whole batch duration)

Score plot with confidence limits



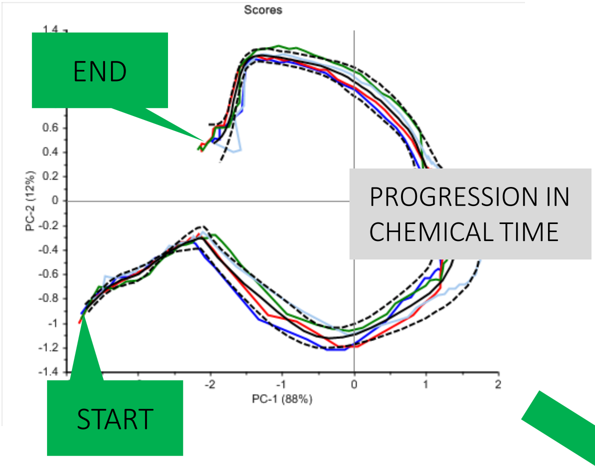
Correlation loadings



The CAMO Strategy

Bring data to life

Offline analysis with The Unscrambler X & Online process monitoring with Process Pulse II



Applications

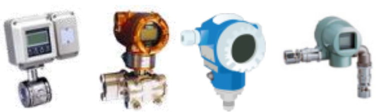
- Fermentation
- Chemical reactions
- Drying
- Mixing

On-line monitoring

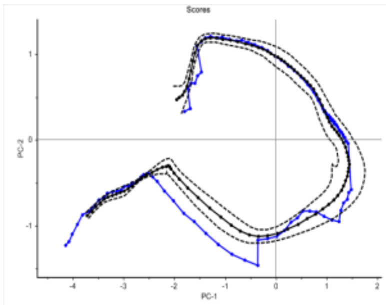
Model repository

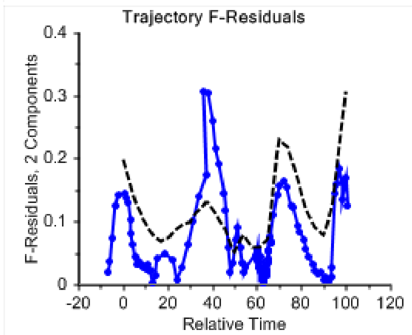
Unscrambler X
Process Pulse

Data in real-time



Graphical presentation and alerts





Solution

- Modeling of batch progression in relative time
- The method is independent of the sampling frequency
- Automatic pretreatment of data
- Dynamic critical limits

CAMO
Bring data to life

Next steps

- www.camo.com/testdrive/
- Demo video, www.camo.com
- Book a live demo, grf@camo.com
- Paper:

Chemometrics and Intelligent Laboratory Systems 149 (2015) 66–72

Contents lists available at ScienceDirect

Chemometrics and Intelligent Laboratory Systems

journal homepage: www.elsevier.com/locate/chemolab

ELSEVIER

CHEMOMETRICS AND INTELLIGENT LABORATORY SYSTEMS

Assumption free modeling and monitoring of batch processes☆

CrossMark

Frank Westad [1], Lars Gidskehaug [1], Brad Swarbrick [1], Geir Rune Flåten [1]

CAMO Software AS, Postboks 1662 Vika, 0120 Oslo, Norway

A R T I C L E I N F O

Article history:
Received 7 January 2015
Received in revised form 31 July 2015
Accepted 27 August 2015
Available online 5 September 2015

Keywords:
Batch processing
Batch modeling
Multivariate modeling

A B S T R A C T

Modeling strategies currently in use for the monitoring of batch processes where multivariate data are available have some limitations, particularly for batches where the true starting or end point are not the same on an absolute time scale, or the batch progression varies among batches. In this paper, a method capturing these differences and allowing modeling and monitoring of batches in relative time is proposed. Using scores from principal component analysis (PCA) models as a feature space the new methodology is better able to handle the challenges usually experienced in batch analysis. The feasibility of the relative time approach is demonstrated using data from a chemical synthesis and a pharmaceutical drying process.

CAMO

Bring data to life

Bring data to life

THANK YOU!

Geir Rune Flåten
grf@camo.com